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Effects of torrefaction on the physiochemical properties of oil palm empty fruit bunches, mesocarp fiber and kernel shell

Khalik M. Sabil [a,*], *Muafah A. Aziz* [a,b], *Bhajan Lal* [a], *Yoshimitsu Uemura* [b]

[a] *Phase Separation Laboratory, Research Centre of CO_2 Capture, Universiti Teknologi PETRONAS,*
Bandar Seri Iskandar, 31750 Tronoh, Perak, Malaysia
[b] *Center of Biofuel and Biochemical, Chemical Engineering Department, Universiti Teknologi PETRONAS,*
Bandar Seri Iskandar, 31750 Tronoh, Perak, Malaysia

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ABSTRACT

In this work, the effects of torrefaction on the physiochemical properties of empty fruit bunches (EFB), palm mesocarp fiber (PMF) and palm kernel shell (PKS) are investigated. The change of properties of these biomass residues such as CHNS mass fraction, gross calorific value (GCV), mass and energy yields and surface structure when subjected to torrefaction process are studied. In this work, these materials with particle size in the range of 355–500 μm are torrefied under light torrefaction conditions (200, 220 and 240 °C) and severe torrefaction conditions (260, 280 and 300 °C). TGA is used to monitor the mass loss during torrefaction while tube furnace is used to produce significant amount of products for chemical analyses. In general, the study reveals torrefaction process of palm oil biomass can be divided into two main stages through the observation on the mass loss distribution. The first stage is the dehydration process at the temperature below than 105 °C where the mass loss is in the range of 3–5%. In the second stage, the decomposition reaction takes place at temperature of 200–300 °C. Furthermore, the study reveals that carbon mass fraction and gross calorific value (GCV) increase with the increase of torrefaction temperature but the O/C ratio, hydrogen and oxygen mass fractions decrease for all biomass. Among the biomass, torrefied PKS has the highest carbon mass fraction of 55.6% when torrefied at 300 °C while PMF has the highest GCV of 23.73 MJ kg^{-1} when torrefied at the same temperature. Both EFB and PMF produce lower mass fraction than PKS when subjected to same torrefaction temperature. In terms of energy yield, PKS produces 86–92% yield when torrefied at light to severe torrefaction conditions, until 280 °C. However, both EFB and PMF only produce 70–78% yield at light torrefaction conditions, until 240 °C. Overall, the mass loss of 45–55% of these biomasses is observed when subjected to torrefaction process. Moreover, SEM images reveal that torrefaction has more severe impact on surface structure of EFB and PMF than that of PKS especially under severe torrefaction conditions. The study concludes that the torrefaction process of these biomass has to be optimized based on the type of the biomass in order to offset the mass loss of these materials through the process and increase the energy value of the solid product.

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* *Corresponding author*. Tel.: +60 53687683 (office), +60 124211974; fax: +60 53755670.
E-mail addresses: khalik_msabil@petronas.com.my, halik98@yahoo.com (K.M. Sabil).

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1. Introduction

Due to depletion of fossil fuels and serious environmental problems associated with the usage of fossil fuels, renewable energy supply is necessary. Biomass is one of the promising renewable energy sources, and it can be utilized as solid, liquid and gas fuels. Growing concerns about the effects of CO_2 emissions from fossil fuels call for sustainable energy sources, such as biomass, because of its carbon neutrality, relative abundance and nonedible [1]. Due to their availability in Malaysia, oil palm residues are considered to be the most abundant biomass and the best options for fuel generation. Malaysia produces about 47% of the world's palm oil supply and can be considered as one of the world's largest producers and exporters of palm oil. Due to the intensive planting and mill operations, Malaysia generates huge quantity of oil palm biomass including oil palm trunks, oil palm fronds, empty fruit bunches (EFB), shells and fibers as residues from harvesting and processing activities. Every year, palm oil industry produces roughly about 17.08 Tg of EFB, 12.9 84 Tg of frond, 8.2 Tg of trunk, 9.66 Tg of mesocarp fiber and 5.3 Tg of kernel shell [2–4]. Each type of these biomasses has different fractions of constituents as summarized in Table 1 [5–7,16].

Due to the huge quantities of biomass generated from the oil palm industry, it will be a waste if the biomass is not properly utilized. In 2005, with production of 15 Tg of oil, energy value of the EFB residue alone is around 125 pJ. This would amount to 1200 M$ for an assumed price of oil of $400 per tonne [8]. The processing of fresh fruit bunches of oil palm results in different types of residues including empty fruit bunch (EFB), mesocarp fiber (PMF) and kernel shell (PKS), which are considered to be harmful wastes to the environment if released untreated [9]. Discharging the biomass waste on the land may lead to pollution and might deteriorate the surrounding environment. Traditionally, EFB is burnt in simple incinerators as a mean of disposal and the ash recycled to the plantation as a fertilizer. Due to air pollution generated from this practice and stringent regulations impose on the burning of biomass residues, EFB is currently used as mulch in the field. This could cut back fertilizer use and improve the soil structure [10]. For last few years, most of the oil palm mills start to use the oil palm residues as fuels in co-generation process of their boiler [11]. Due to their characteristics, some of these resources have to be pretreated before they can be utilized as fuel. Therefore, pretreatment process is required to improve the energy yield of biomass.

Table 1 – Oil palm chemical composition analysis.

Component (W_B on biomass raw sample)

Components	EFB	PMF	PKS
Holocellulose	0.3530	0.3180	0.2230
Cellulose	0.3830	0.3440	0.2080
Lignin	0.2221	0.2527	0.5070
Ash	0.0167	0.0350	0.0104
Moisture	0.6700	0.3700	0.1200

Refs. [5–7,16].

Several challenging properties of biomass including high moisture, low energy density, hygroscopic nature, low heating value and soot formation during combustion limit its usage as a resource for the bio-energy [12–14]. The utilization of the raw biomass during thermo-chemical conversion processes such as pyrolysis and gasification entailed several problems. In most cases, biomass has relatively high moisture value [15–18]. In the case of EFB, the moisture can be as high as 60–67% where as PMF and PKS have 30–37% and 12–20% of moisture, respectively [19–21]. This leads to the difficulty in handling and transportation of these biomass residues [15,22,23]. Secondly, due to its fibrous and tenacious nature, biomass is difficult to grind into small homogenous particle. This poor grindability issue can cause serious problems especially when the biomass is used in the pulverized system such as co-firing with coal [13,15,18]. Moreover, very high load of untreated biomass is required to generate an equivalent amount of energy from coal due to its relatively low carbon [13]. Finally, raw biomass is also characterized as having low heating value, high O/C ratio and low energy density [15–18]. All these drawbacks contribute to the difficulty of utilization of untreated biomass.

In order to overcome these limitations, a pre-treatment process is often necessary to improve the quality of biomass. One of the processes that are often applied is torrefaction process [15–18]. Torrefaction is a thermal treatment process where biomass is heated within a temperature range of 200–300 °C under an inert atmospheric condition. The products of torrefaction consist of dark color solid, condensate including moisture and acetic acid, and gases which are mainly CO_2, CO and small amounts of CH_4 and H_2 [13,16]. The torrefied solid has lower moisture and O/C ratio with higher carbon mass fraction and calorific value compared to the raw biomass. The low moisture can prevent biodegradation, thus can increase the storage duration of the treated solid. The higher carbon and calorific value are contributing to the increase in the energy yield of the torrefied solid. Besides, the tenacious and fibrous nature of biomass becomes more brittle once torrefied, which improves the grindability of the solid [13,16,17,24].

One important parameter that influenced the torrefaction process is temperature. Many researchers have reported that torrefaction temperatures for lignocellulosic biomass range from 250 to 300 °C [2–4,22–36]. Aziz et al. have reported that torrefaction of oil palm biomass residues is affected by the chemical composition and decomposition temperature [25]. Chen et al. showed that torrefaction at 230 °C has a relatively small impact on the decomposition of basic biomass components. However, as the temperature is increased to 260 °C, a significant amount of hemicellulose decomposed, and at 290 °C, a larger amount of hemicellulose and cellulose decomposed [26]. In addition, Yan et al. reported that an increase in the torrefaction temperature will result in a decrease in mass fraction and an increase in densification of torrefied biomass, which produce a solid with an increase of C and a decrease of O and volatiles [27]. Similarly, Deng et al. also reported that an increase of torrefaction temperature leads to a decrease in solid bio-char yield and an increasing yield in volatile matters including liquid and noncondensable gases

[23]. Chen and Kuo pointed out that hemicellulose is influenced by light torrefaction, whereas cellulose and lignin are hardly affected at temperature below than 240 °C [22]. Presently, most of reported studies on torrefaction are focused on woody and grass biomass [16,17,28–30] and very few reported data on oil palm biomass residues are available [2–4].

In the present study, our main objective is to investigate the torrefaction behavior of EFB, PMF and PKS and its influence on their physiochemical properties such as GCV, O/C ratio, mass loss distribution, changes on the surface structure, mass and energy yields. It is our believe that the findings of this study will be beneficial for utilization of these biomass residues in energy production especially in co-generation process of palm oil mills. Moreover, the physiochemical properties of torrefied biomass will provide some indications on the suitability of these materials for further thermochemical processes such as pyrolysis or gasification.

2. Experimental

2.1. Materials

In this study, EFB, PMF and PKS of fresh fruit bunch (FFB) are studied as shown in Fig. 1. The fresh fruit bunches are harvested from 10 to 20 years old oil palm trees (*Elaeis guineensis*) (height 12 m, diameter 30–45 cm). The EFB, PMF and PKS are collected from a palm oil mill processing plant where the FFB is used as the feedstock. The plant is located at FELCRA Nasaruddin plantation in Bota, Perak province (4° 21′ 10.50″ North, 100° 54′ 20.33″ East), Malaysia. They are collected after the oily flesh of the fruit has been removed for further processing. Several operational units are involved in the separation process of EFB, PMF and PKS from FFB as shown in Fig. 2. EFB residue is generated at the thresher, where fruits are removed from FFB. PMF is generated at the nut/fiber separator where as PKS is generated from the shell/kernel separator. Since each one of the biomass residue can be collected at different stages during the processing stages, it is easy to treat each one of them separately and it does not involve any additional cost.

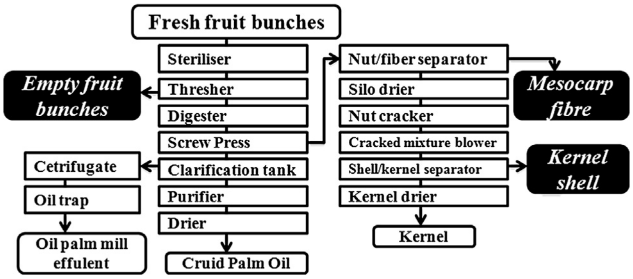

Fig. 2 – Simplified oil palm mill operation for separation of EFB, PMF and PKS.

After collection, all the materials are oven dried at 105 °C for 24 h before being stored in air-tight container and used for experimentation without any pre-treatment [31,36]. The predrying is necessary to avoid further biodegradation of the sample during storage since the moisture mass fraction of the fresh EFB, PMS and PKS is relatively high as explained earlier [19–21]. Moreover, the pre-drying is used to simulate the industrial practice of sun-drying the materials before storage. After the drying procedure, the raw materials are grinded and sieved to particle size in the range of 355–500 μm. This size range is chosen as it has been shown that particle sizes below 500 μm did not exert any significant influence on the rate of torrefaction process [23,32]. Then, the prepared raw materials are placed in plastic bags and stored in a desiccator at room temperature until the analyses are carried out.

2.2. Lignocellulosic analysis of the raw materials

In this work, the lignocellulosic analysis is conducted according to the conventional adaptation method [33]. Although there are other methods available such as HPLC and NREL, the adaptation method is chosen since it can give the exact amount of hemicellulose where method such as NREL fails to provide the hemicellulose fraction accurately. This adaptation method comprises of determination of holocellulose (sum of hemicellulose and cellulose), α-cellulose (cellulose) and lignin. For the determination of holocellulose, 3 g of biomass is

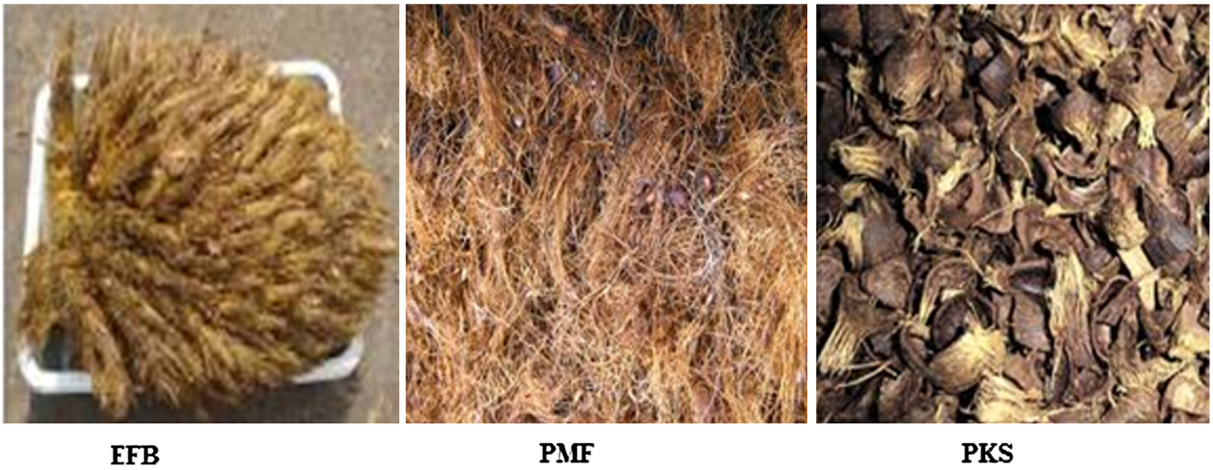
EFB PMF PKS

Fig. 1 – Photos of three type of oil palm raw biomass namely EFB, PMF and PKS.

treated in 0.12 L of 3% *v*/*v* chlorine water and is left about 5 min. Then, the solution is filtered by using vacuum filtration and the filter cake is washed with 0.25 L of 5% *v*/*v* H_2SO_3 solution and 0.50 L of distilled water. Then, the produced filter cake is transferred into 0.10 L beaker and 0.05 L of 20.48 kg m^{-3} Na_2SO_4 solution is added into the beaker. The beaker is left in boiling water bath for 30 min and after completion, the solution is filtered again. After that, the filter cake is transferred into 0.10 L beaker and 0.02 L of 1.00 kg m^{-3} $KMnO_4$ solution is added and the solution is left for 10 min. Next, 3% *v*/*v* H_2SO_3 solution is added to neutralize the solution. The mixture is then transferred into 0.5 L beaker that is initially filled with 0.20 L of distilled water. The beaker is placed in boiling water bath for 1 h. Then, the solution is filtered and the cake is washed with 0.50 L of hot distilled water and follows by ethanol. Finally, the filter cake is dried at 105 °C for 4 h and weighed. The residue is the sum of hemicellulose and cellulose fractions.

In the second part, approximately 2.0–2.5 g of the residues from the first part is placed into 0.20 L conical flask and is treated with 0.12 L of 212 kg m^{-3} NaOH solution. The solution is constantly stirred by using a magnetic stirrer at ambient temperature for 24 h. After the process is completed, the solution is filtered with distilled water. Finally, the filter cake is dried at 105 °C for 4 h and weighed. The left residue from this part is considered as the cellulose mass fraction. The hemicellulose mass fraction is calculated as the difference between the holocellulose value (first part) and α-cellulose value (second part).

In the third part, to determine lignin mass fraction, 1 g of biomass is placed into 0.1 L of beaker and 0. 01 L of 75% *v*/*v* H_2SO_4 solution is added and the solution is treated at 20 °C in a water bath for 4 h. Then, the solution is transferred into 1 L conical flask and 0.56 L of distilled water is added. The solution is left to reflux for 4 h in a boiling water bath. Once completed, the residue is filtered and washed with 0.5 L of distilled water. Finally, the filter cake is dried at 105 °C for 4 h and weighed. The residue is taken as the lignin fraction of the biomass. For each biomass sample, three measurements are conducted and the average value from these measurements is reported.

2.3. *Torrefaction process*

Torrefaction process is carried out with 2 g of biomass by using EXSTAR TG/DTA 6300-SII thermogravimetric analyzer (TGA). Nitrogen is used as a carrier gas at flow rate of 0.1 L min^{-1} with fixed heating rate of 10 °C min^{-1}. Temperature programme consisting of a dynamic and isothermal heating period is used for torrefaction process. First, the TGA temperature is raised from room temperature to 50 °C. Once the TGA temperature is constantly stable, temperature is further raised until 120 °C and maintained for 10 min. Then, the TGA temperature is raised to each desired final torrefaction temperatures of 200, 220, 240, 260, 280 and 300 °C, accordingly. Once the TGA reached to the final torrefaction temperature, the biomass is continuously torrefied for 2 h. Due to the limited amount of torrefied biomass produced from TGA for analyses, the exact procedure is replicate in a tube furnace for mass production of the torrefied material. For the TSH17/75/450-2416-2116 tube furnace, 2 g of raw biomass is used. The same temperature programme procedure is applied.

2.4. *Solid characterization*

The chemical and physical characteristics of the solids are verified using elemental analysis, calorific value and scanning electron microscopy (SEM). All the analyses are carried out for both the raw material and torrefied solids. The elemental analysis is performed using LECO CHNS elemental analyzer to measure the elemental value of carbon, hydrogen, nitrogen and sulfur. The calorific values are determined using IKA-WERKE C5000 bomb calorimeter. The measured GCV is based on the high heat value (HHV) which includes the heat of condensation of water vapor produced in the combustion. In this work, for each sample, three measurements are conducted and the average value from these measurements is reported. Surface morphology of torrefied biomass is determined using scanning electron microscope Model 1430 Germany.

3. Results and discussion

3.1. *Mass loss distribution by TGA*

During torrefaction process, the mass loss distributions for all types of biomass have been monitored by TGA as shown in Figs. 3–5. It can be observed that the mass loss is significantly influenced by torrefaction temperature. Chen and Kuo [22] have classified two different torrefaction conditions, at or under 240 °C as light torrefaction and at or above 275 °C as severe torrefaction, to investigate the influence of torrefaction temperature on the properties of torrefied biomass. In this work, we follow these classifications. Figs. 3–5 clearly show that the torrefaction process of oil palm biomass can be divided into two main stages. The first stage is mainly due to the dehydration process where the remaining moisture of the pre-dried biomass is released from the biomass at the temperature below than 105 °C. This moisture may be absorbed from the atmospheric condition during storage since the atmospheric relative humidity in Malaysia is generally higher than 80% [34]. During this stage, the mass reduction is observed to be within the range of 3–5% for all biomass.

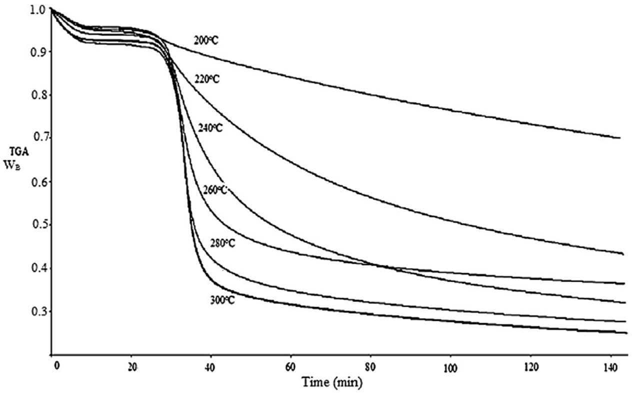

Fig. 3 – Mass loss distributions for EFB size of 355–500 μm.

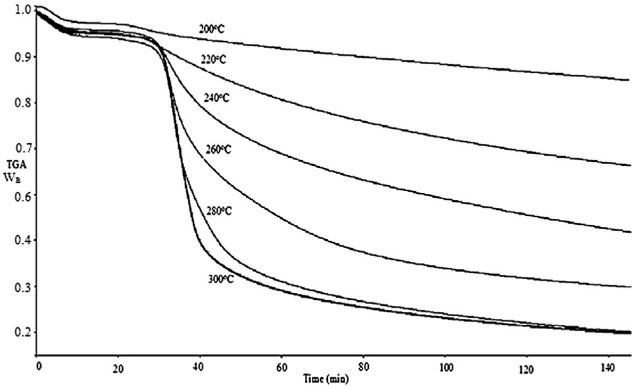

Fig. 4 – Mass loss distributions for PMF size of 355–500 μm.

In the second stage, the decomposition reaction is took place at 200–300 °C, where the significant mass reduction has been observed for all types of biomass. During these stages, the mass reduction is within the range of 45–55%, depending on the final torrefaction temperature and type of biomass used. These two stages are clearly demonstrated through the mass loss distributions for each torrefied biomass as shown in Figs. 3–5. Besides, the slopes of the curves are considered as the decomposition rate of the biomass during torrefaction process. From these figures, it is found that the slopes of curves are increased as the torrefaction temperature increased. Thus, it is concluded that the decomposition rate is higher at higher torrefaction temperature, especially at temperature above 260 °C. However, after 140 min of torrefaction time, the curves of mass loss distributions for each types of torrefied biomass are remained stable which indicating that the decomposition of the biomass had been almost completed. The characteristics of the mass loss during the torrefaction process are in agreement with results of other studies for different type of biomass [21,30,35].

The remaining mass for each types of biomass for every final torrefaction temperatures is listed in Table 2. It is found that after the biomass undergone light torrefaction conditions, 41.45% of EFB, 49.06% of PMF and 56.98% of PKS are remained at the temperature of 240 °C. However, after severe torrefaction conditions, only 28.80% of EFB, 32.76% of PMF and 36.42% of PKS are remained at 300 °C.

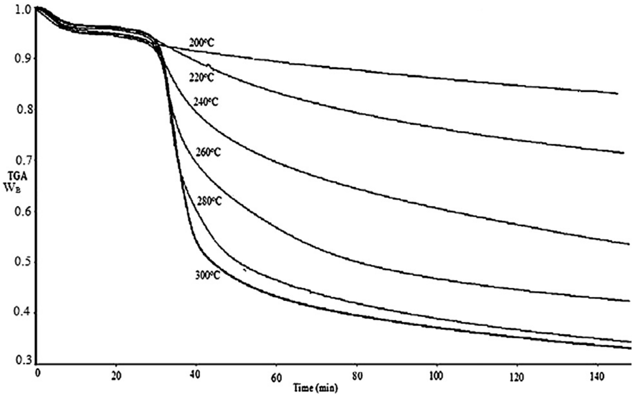

Fig. 5 – Mass loss distributions for PKS size of 355–500 μm.

Table 2 – Biomass mass remained (W_B) for every torrefaction temperatures.

Temperature (°C)	Biomass mass remained (W_B) at size of 355–500 μm		
	EFB	PMF	PMF
200	0.7456	0.8818	0.8126
220	0.4822	0.7529	0.6472
240	0.4226	0.4731	0.5832
260	0.3621	0.4642	0.3734
280	0.3123	0.3949	0.3552
300	0.2986	0.3214	0.3642

The highest mass loss of EFB may be attributed to the higher mass fraction of hemicellulose in EFB compared to both PMF and PKS as reported in the lignocellulosic analysis presented in Table 3. It is well-known that high mass fraction of hemicellulose can enhance the decomposition rate during torrefaction process [24,37]. Moreover, the hollow structure of EFB as shown in Fig. 9 also may contribute the faster rate of decomposition. Other than that, the study had also revealed that the PKS is the most difficult to be decomposed during torrefaction. High lignin mass fraction of PKS had contributed in lower decomposition rate as seen in Table 3. Hence, the mass remained for PKS is the highest after 300 °C, compared to both PMF and EFB. Therefore the decomposition rate of oil palm biomass is affected by the torrefaction conditions and composition of hemicellulose, cellulose and lignin of the biomass.

3.2. *Elemental analysis and calorific value of biomass*

Table 4 presents the CHNS mass fraction [25] and GCV for the raw and torrefied EFB, PMF and PKS. The values obtained from elemental analysis for EFB in this work are comparable with reported literature data [36].The ultimate analysis shows that torrefaction increases the carbon fraction of torrefied solid, whilst decreases the H and O fractions. The decrease of H and O in the torrefied materials may be attributed to the decomposition reactions that take place during the process to produce volatile gases. Moreover, there is an increment in nitrogen as the torrefaction temperature increase for all biomass materials with PKS has the lowest mass fraction of nitrogen in comparison to PMF and EFB. Since the percentages of nitrogen higher than 0.6% can cause problems related to the

Table 3 – Lignocellulosic analysis of the raw EFB, PMF and PKS.

Components	Oil palm biomass size of 355–500 μm (W_B)					
	EFB[a]	EFB[b]	PMF[a]	PMF[b]	PKS[a]	PKS[b]
Hemicellulose	0.2446	0.3689	0.2303	0.3457	0.1824	0.2410
Cellulose	0.4906	0.4002	0.4404	0.3750	0.3320	0.2208
Lignin	0.2648	0.2309	0.3294	0.2793	0.4856	0.5382

a In the present study.
b Ref. [2].

emissions of nitrogen oxides [38–40], all of the treated materials may pose the problem once use as fuels especially when they have been torrefied in severe condition. As the biomass are torrefied from light to severe torrefaction conditions, the alterations have been occurred in the elemental composition and GCV, especially at the torrefaction temperature above 260 °C. In particular, the GCV increased with the torrefaction temperature whilst the O/C ratio decreased. These results seem to be in the agreement with the literature [13,14,17].

Torrefaction temperature affects the gross calorific value of the studied biomass as its value increased with the increase of the torrefaction temperature. The increasing of the GCV is mainly related to the increasing of the carbon fraction in the torrefied biomass [13,17]. Also, the increase of GVC is influenced by the type of biomass. For example, at 240 °C, both PMF and PKS have slightly higher GCV with 20.09 MJ kg^{-1} and 20.03 MJ kg^{-1}; respectively compared to EFB with only 19.38 MJ kg^{-1}. However, at 300 °C, PMF had the highest GCV of 23.73 MJ kg^{-1} whereas EFB and PKS have GCV of 22.42 MJ kg^{-1} for EFB and 22.86 MJ kg^{-1}, respectively. These results show that the GVC of these biomass residues can be improved from approximately 18 MJ kg^{-1} to more than 22 MJ kg^{-1}

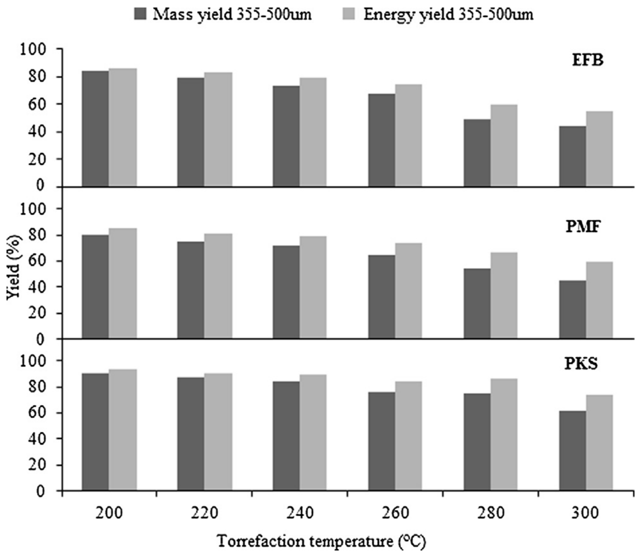

Fig. 6 – Mass and energy yields for EFB, PMF and PKS as a function of torrefaction temperatures.

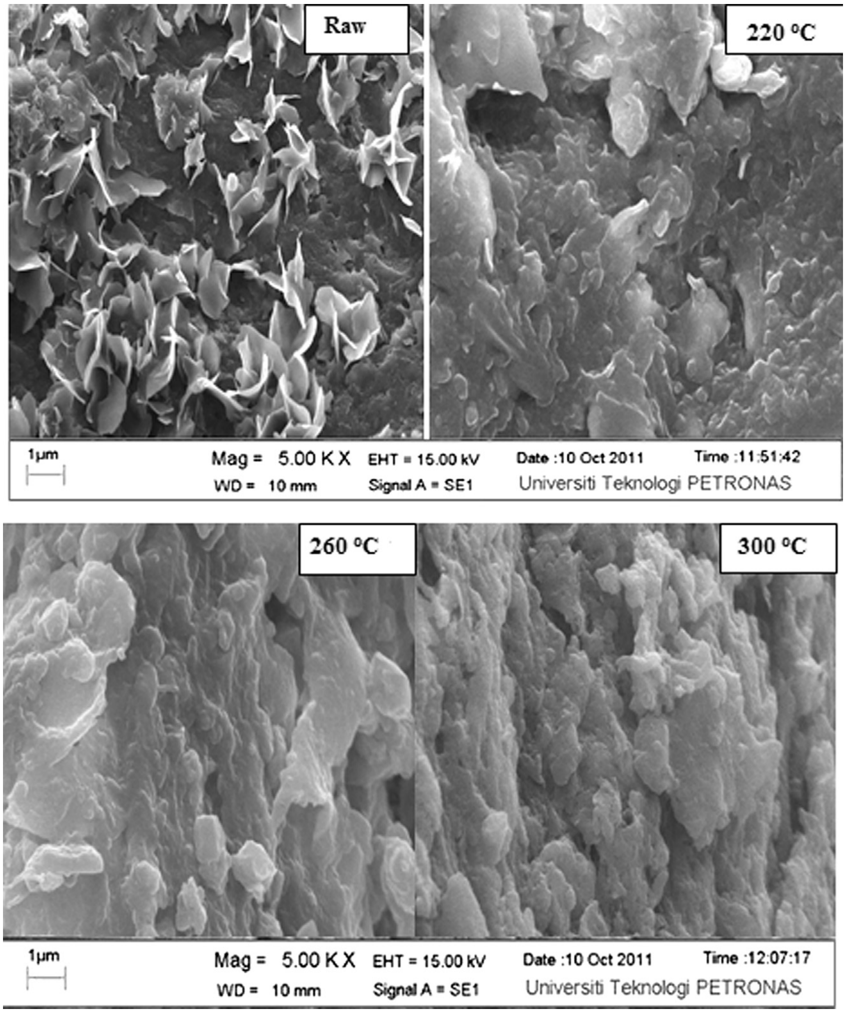

Fig. 7 – SEM images for raw and torrefied EFB for size of 355–500 μm.

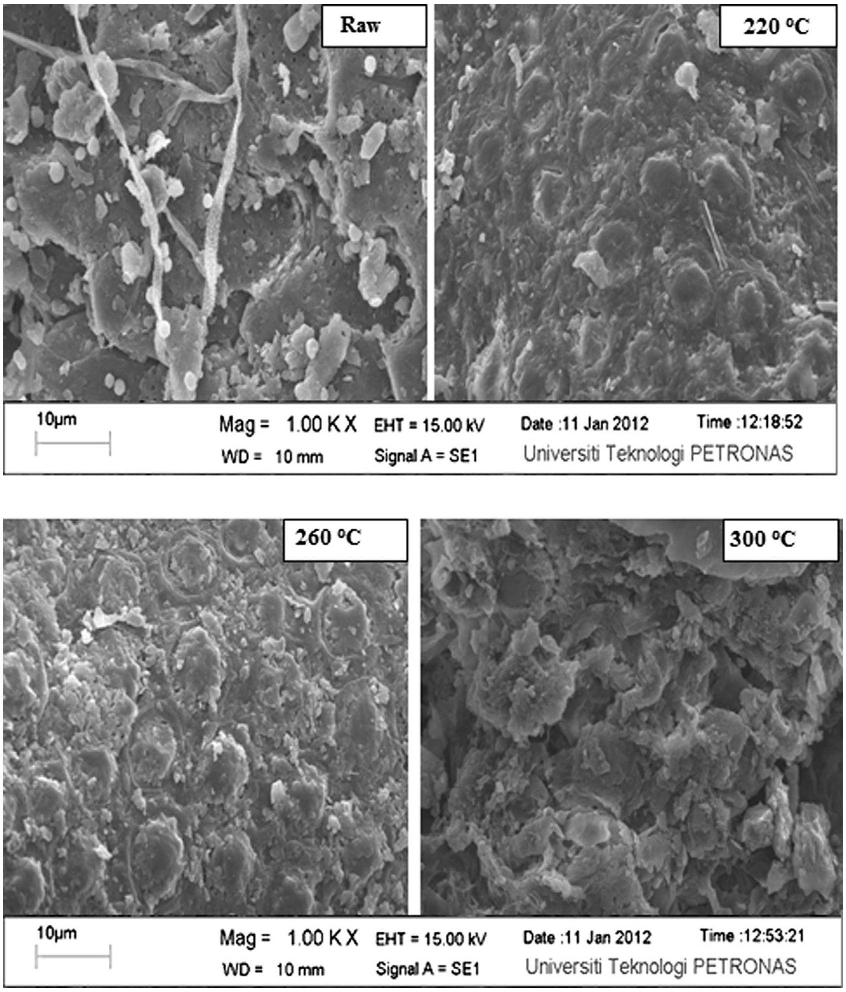

Fig. 8 – SEM images for raw and torrefied PMF for size of 355–500 μm.

depending on the type of biomass and the selected temperature.

In this work, the O/C ratio is reported in mass ratio. The O/C ratios of EFB, PMF and PKS are in the range of 1.052–1.107. The O/C ratio obtained for EFB in this work is comparable with reported literature data of 1.13 [36]. Due to the decomposition of the biomass and elimination of volatile matter during torrefaction process, the oxygen mass fraction of the torrefied products will be lower. Therefore, the O/C ratio of all torrefied products obtained is lower than that of the raw biomass and these results are in line with literature [13,17]. As the torrefaction temperature increased, the O/C ratio of EFB, PMF and PKS is gradually reduced as more volatile matter being released as a result of the continuous decomposition process. At 300 °C, the O/C ratios of the torrefied PKS, PMF and EFB are 0.606, 0.805 and 0.828, respectively.

3.3. *Mass fraction and energy yield*

In biomass energy, torrefaction aims for the production of a fuel that is having improved properties compared to the original biomass. However, this should be achieved without losing too much chemical energy due to the release of volatile products during the treatment process. Therefore, the mass fraction and energy yield are considered as the crucial parameters in evaluating a torrefaction process. Moreover, GCV of biomass is an important property of a solid fuel since it is an expression of energy mass fraction that is released when the solid is burnt. The obtained results of the mass fraction and energy yield for EFB, PMF and PKS are presented in Fig. 6 whereas the measured GVC is reported in Table 4. In the present study, the GCV of torrefied biomass is measured in the high heat value (HHV) basis. Both of the mass fraction and energy yield (Y_{mass} and Y_{energy}) of the torrefied biomass are defined according to Eqs. (1) and (2) [13,17].

$$Y_{\text{mass}}(\%) = (\text{mass after torrefaction}/\text{mass before torrefaction}) \times 100 \tag{1}$$

$$Y_{\text{energy}}(\%) = Y_{\text{mass}} \times \left(\text{HHV}_{\text{torrefied biomass}}/\text{HHV}_{\text{raw biomass}}\right) \tag{2}$$

Fig. 6 shows the mass fraction and energy yields decreased steadily as the torrefaction temperature increased. The obtained results are consistent with the previous works

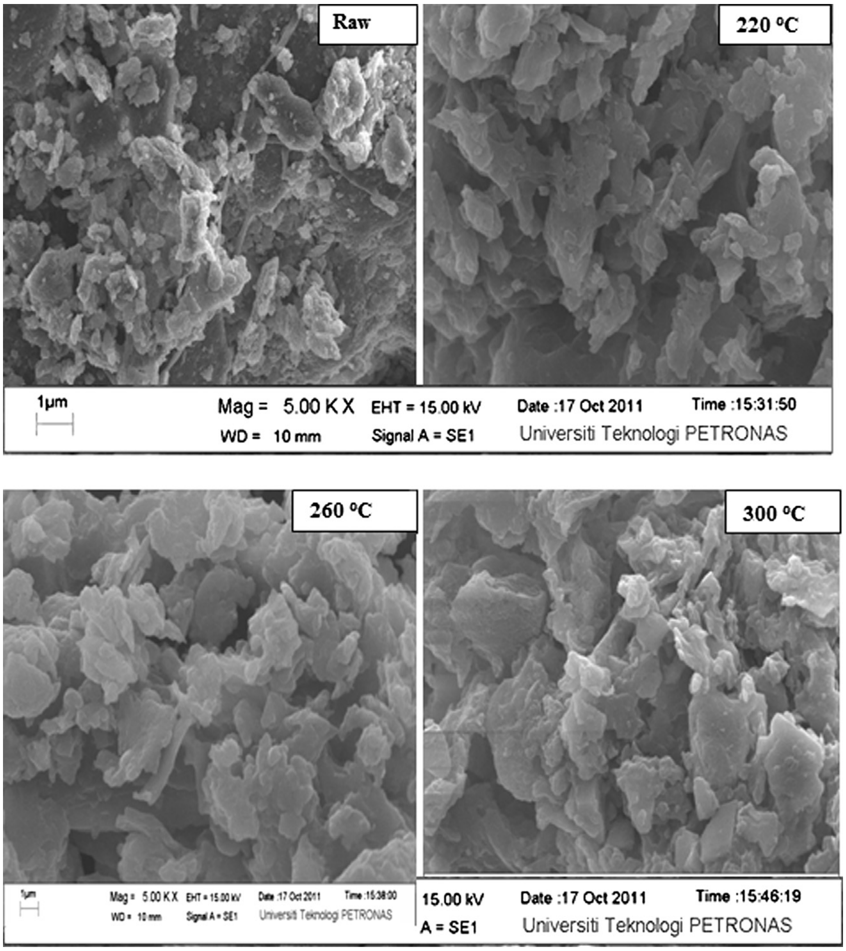

Fig. 9 – SEM images for raw and torrefied kernel shell for size of 355–500 μm.

Table 4 – Elemental analysis and gross calorific value for torrefied EFB, PMF and PKS.

Biomass	Temperature (°C)	Ultimate analysis (W_B)					O/C ratio	GCV (MJ kg^{-1})
		C	H	N	S	O		
EFB size 355–500 μm	Raw	0.4460	0.0548	0.0037	0.0018	0.4937	1.107	18.04
	200	0.4749	0.0479	0.0116	0.0015	0.4641	0.977	18.44
	220	0.4833	0.0472	0.0125	0.0008	0.4562	0.944	18.90
	240	0.4927	0.0466	0.0127	0.0008	0.4472	0.908	19.38
	260	0.4963	0.0451	0.0127	0.0007	0.4453	0.897	19.89
	280	0.4984	0.0453	0.0152	0.0010	0.4401	0.883	22.18
	300	0.5156	0.0419	0.0143	0.0011	0.4272	0.828	22.42
PMF size 355–500 μm	Raw	0.4691	0.0544	0.0063	0.0011	0.4692	1.000	18.16
	200	0.4789	0.0492	0.0123	0.0007	0.4590	0.958	19.34
	220	0.4765	0.0473	0.0134	0.0006	0.4622	0.970	19.67
	240	0.4787	0.0468	0.0143	0.0014	0.4589	0.959	20.09
	260	0.5050	0.0427	0.0156	0.0009	0.4359	0.863	20.75
	280	0.5109	0.0391	0.0162	0.0007	0.4332	0.848	22.05
	300	0.5150	0.0378	0.0165	0.0004	0.4302	0.835	23.73
PKS size 355–500 μm	Raw	0.0450	0.0621	0.0042	0.0018	0.4819	1.071	18.91
	200	0.0459	0.0599	0.0087	0.0003	0.4723	1.029	19.48
	220	0.4684	0.0578	0.0091	0.0002	0.4645	0.992	19.78
	240	0.4680	0.0563	0.0089	0.0004	0.4663	0.996	20.03
	260	0.4820	0.0506	0.0097	0.0013	0.4561	0.946	20.83
	280	0.5520	0.0491	0.0089	0.0003	0.3901	0.707	21.91
	300	0.5560	0.0471	0.0093	0.0008	0.3873	0.697	22.86

[13,29,34] where EFB shows the highest decrease in these yields compared to PMF and PKS. Under light torrefaction conditions, the mass fraction for EFB, PMF and PKS is 73%, 71% and 84%, respectively. However, the mass reduces drastically to 43% for EFB, 45% for PMF and 61% for PKS at 300 °C.

Moreover, results show that the energy yield of EFB, PMF and PKS decreased as the torrefaction temperature increased. The energy yield obtained from EFB is in the range of 55–89% at 200 and 220 °C. Meanwhile, PMF produces 57–85% energy yield 200 and 220 °C. In general, the lower energy yield produced of both EFB and PMF is mainly caused by the poor mass fraction [32]. The produced energy yield of PKS is in the range of 72–93%, with the highest obtained value is at 200–240 °C. In comparison with the energy yields of both EFB and PMF, PKS always has higher energy yield either after light or severe torrefaction conditions. Thus, our study reveals that the energy yield of PKS is the least affected by the torrefaction temperature compared to both EFB and PMF.

The energy yield for all biomass is drastically reduced to 56–58% for EFB and PMF while 73% for PKS at 300 °C. This observation may be caused by further decomposition of hemicellulose and cellulose in PKS. Therefore, it is suggested that EFB and PMF should be torrefied under light torrefaction conditions to preserve the energy yield above than 78%. Meanwhile, PKS can be torrefied under severe torrefaction conditions up to 280 °C in order to obtain energy yield around 80–86%. Thus, this study concludes that the mass and energy yield are depending on the torrefaction temperature and biomass types. In addition, torrefaction oil palm biomass at 300 °C is not recommended since it produces low energy yield which is less than 70% and low mass fraction which is less than 45%.

3.4. *Microscopic observation*

Figs. 7–9 show that the changes on the surface structure of torrefied biomass due to torrefaction process can be observed in the SEM images for EFB, PMF and PKS. These images are taken for particle size of 355–500 μm at 220, 260 and 300 °C, respectively. The surface structure of raw EFB shows a clearly sharp edge which is not present on the surface of both raw PMF and PKS. This study suggests that the presence of sharp edges on the surface of raw EFB may be related with the grinding effect. Furthermore, the presence of pores is observed on the surface of both raw EFB and PMF but not on the surface of raw PKS. Thus, present investigation suggests that the presence of the pores on the surface of both raw EFB and PMF has verified they are fibrous materials.

The changes on the surface structure for each torrefied biomass are observed as compared to the raw biomass when they subjected to torrefaction process. These observations are in the good agreement with literature [13,37]. It is found that the internal structure of both EFB and PMF is the most affected as observed in Figs. 7–9. Moreover, the impact of the torrefaction temperatures on the internal structure of EFB and PMF is clearly observed especially at 260 and 300 °C. These observations show that the internal structure of both EFB and PMF is completely decomposed under severe torrefaction conditions. The presence of the sharp edges on the surface of raw EFB has almost flattened and disappeared while the presence of pores on the surface of raw PMF has diminished.

As reported by the lignocellulosic analysis in Table 3, both EFB and PMF possess higher amount of hemicellulose as compared with PKS. Thus, they are presumed to start having decomposition at lower temperature of 150 °C while at higher temperature of 300 °C, the severe decomposition has occurred on the internal structure both of them. Therefore, the changes of internal structure of both EFB and PMF are affected by decomposition temperature of hemicellulose. However, it is found that the internal structure of torrefied PKS is the least affected as Fig. 9 shows. The observations at 260 and 300 °C have showed that the internal structure of torrefied PKS does not differ so much, just slightly changes occurred. Present study suggests that PKS demonstrates less impact of decomposition to the internal structure due to its high lignin mass fraction as reported by the lignocellulosic analysis in Table 3. The wide range of the decomposition temperature of lignin has started at 250 °C until 500 °C. The decomposition of PKS has just started at 260 °C while at 300 °C, the decomposition is still not completed. Therefore, the changes of internal structure of torrefied PKS are not clearly observed and are least affected by decomposition temperature of lignin. Our study concludes that the influence of torrefaction temperature on the decomposition of the internal structure of both torrefied EFB and PMF is more prominent than PKS. In addition, the decomposition temperatures of the hemicellulose affect the decomposition of both EFB and PMF while the decomposition temperatures of the lignin affect the decomposition of PKS.

4. Conclusions

In this conclusion, this study reveals that the carbon mass fraction and GCV of torrefied oil palm biomass residues increase as the torrefaction temperature increase. Among the biomass studies, PKS has the highest carbon fraction with 58.7% while PMF has the highest GCV with 23.73 MJ kg^{-1}. However, as the torrefaction temperature increases, the O/C ratios, hydrogen and oxygen fractions decrease. In addition, both EFB and PMF produce lower mass fraction compared to PKS. The energy yield of PKS can be maintained within the range of 86–92% from light to severe torrefaction conditions, until 280 °C. However, the energy yield of both EFB and PMF is within the range of 70–78% at light torrefaction conditions, until 240 °C. Moreover, the SEM images verify that the impact of torrefaction on the internal structure of both EFB and PMF is more significant than PKS under severe torrefaction conditions. In general, the study concludes that these differences properties and characteristics of torrefied oil palm biomass are mainly dependant to the torrefaction conditions and the type of biomass residues.

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